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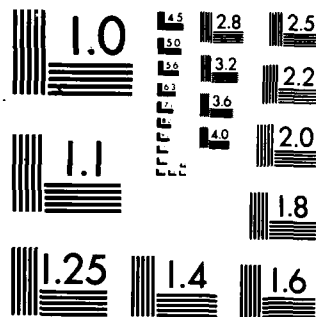
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| <p>An ion beam deposition system was purchased and utilized for research on magnetic materials and devices for high density magnetic information storage. Initial work was carried out on the deposition of permalloy and the deposition of magnetic oxides.</p> <p>The work on permalloy revealed that ion beam deposited materials generally had smaller grain size and lower coercivity than R.F. sputtered materials. It was also found that the grain size and coercivity of ion beam deposited materials increased if a second ion gun were used to bombard the substrate during the deposition process. This work is being continued with support from other sources.</p> <p>The work on magnetic oxides was begun with the deposition of cobalt ferrite. X-ray diffraction measurements indicate the material deposited was amorphous and exhibited</p> |  |  |  |  |                  |
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a hard axis of anisotropy perpendicular to the plane of the film. Coercivity was less than 200 Oe in the plane of the film. This work is also being continued with support from other sources.



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**Fabrication of Material and  
Devices for Very High Density  
Information Storage**

GRANT AFOSR-85-0100

**FINAL SCIENTIFIC REPORT**

by

**M. H. Kryder, C.-H. Huang, C. Bowman and D. Thuel  
Department of Electrical and Computer Engineering  
Carnegie Mellon University  
Pittsburgh, PA 15213**

Under grant AFOSR-85-0100 a dual gun ion beam deposition system was purchased. This system has been installed and used to carry out research on (1) high permeability NiFe alloys and (2) the ion beam deposition of magnetic oxides. Both of these projects relate to work supported by on-going grant AFOSR 84-0341, but also receive support from industrial organizations.

The work on NiFe relates to the fabrication of thin film magnetoresistive detectors for magnetic bubble devices. It is believed that superior detectors may result from this process as compared to conventional sputtering or evaporation. The work on magnetic oxides, though directed at magneto-optical recording materials, could, if successful, also result in the deposition of bubble garnets by ion beam deposition rather than liquid phase epitaxy. This would significantly reduce cost and improve the uniformity of the very thin garnets used in the highest density bubble devices.

The initial work carried out with the ion beam deposition system on these projects has shown some unique advantages, over conventional sputtering, and it is anticipated that our use of ion beam deposition will increase even more in the future. It is expected that this system will continue to be extensively used for at least the next five to ten years. The research carried out on these two projects is summarized below.

**Comparison of Microstructure for NiFe  
Thin Films Prepared via Ion Beam and  
RF Diode Sputtering**

**Introduction:**

Ion-beam deposition is being investigated as a means for producing NiFe thin films suitable for use as magnetoresistive bubble detectors and thin-film recording heads. This technique offers some distinct advantages over the conventional evaporation and radio-frequency (RF) sputtering methods. A study is being done comparing the macroscopic magnetic properties and microstructure for NiFe thin films deposited via RF sputtering and ion beam methods. The comparison involves optimizing the films' magnetic properties for each process while observing changes in the microstructure. In this way insight can be gained into the effect of process parameters on the structure and properties of NiFe thin films.

The first stage of the experiment is concerned with the parameters affecting the size and orientation of the films' crystallites. A study was made into the effect of film thickness on grain size, and into the effects of negative RF substrate bias and the effects of a second ion beam directed at the substrate on the grain size and magnetic properties.

### Equipment

The RF sputtering system is a Perkin-Elmer 2400 RF diode system with a 4" CTI high-vacuum cryo pump. Background chamber pressure is 10 mtorr argon; RF power is 500W and target bias is -2.3kV with respect to the plasma. The ion beam system is a Commonwealth Scientific with dual 4" Kaufman type ion sources, rotatable substrate stage, and a 10" CTI high-vacuum cryo pump. Background chamber pressure is 0.15 mtorr argon, deposition beam energy of 1.3keV, total deposition beam current ranging from 100 to 150mA, and average deposition beam current density of  $0.75\text{mA}/\text{cm}^2$ . For the resputtering source, beam energy is 0.5keV, total beam current 11mA, and average current density is  $0.06\text{mA}/\text{cm}^2$ . The average deposition rate for the RF system is 300 Angstroms/minute compared with 100 Angstroms/minute for the ion beam system. Substrates are glass, mounted parallel to the target on water-cooled stages for both systems. The substrate is rotated at a rate of 1 rpm during deposition in the ion beam system for uniform coating. Transmission electron microscope (TEM) samples are deposited on 1/8" diameter carbon coated Cu grids. Film thickness ranges from 50 to 6000 Angstroms.

### Discussion and Conclusions:

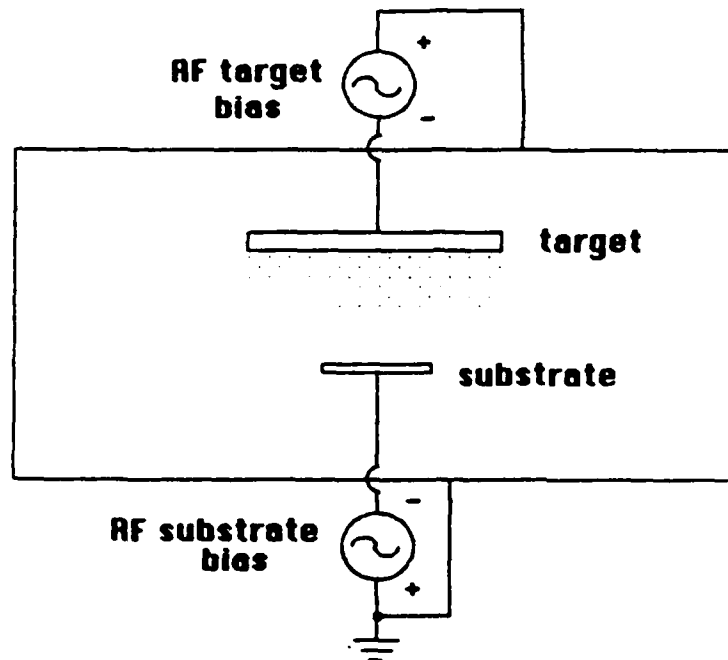
From the data collected we find the grain size of the NiFe films to be independent of the thickness over a 270 to 680 Angstrom range (Fig. 1). Apparently, no significant annealing takes place during the increased deposition time. The ion beam deposited films exhibit smaller grains (50 to 100 Angstroms) than those by the RF process (300 to 400 Angstroms), assumed to be due to the lower substrate temperature during deposition (Fig. 2). Because the plasma generating the beam ions is isolated from the deposition chamber, the substrate is not subjected to bombardment from high temperature secondary electrons, remaining cooler than in the RF process. Grain size increases to 100 to 200 Angstroms when the resputtering beam is directed onto the substrate during deposition (Fig. 2), however, no change in size is seen in the RF sputtered films with the -50V table bias applied. The introduction of the resputtering beam is an additional source of substrate heating, which increases the surface mobility of the arriving atoms, and therefore this result is as expected.

The crystallites show a random orientation for both the ion beam films deposited without resputtering and the RF films with and without the RF table bias. With resputtering present in the ion beam deposition, the crystallites assume a predominantly  $\langle 111 \rangle$  orientation.

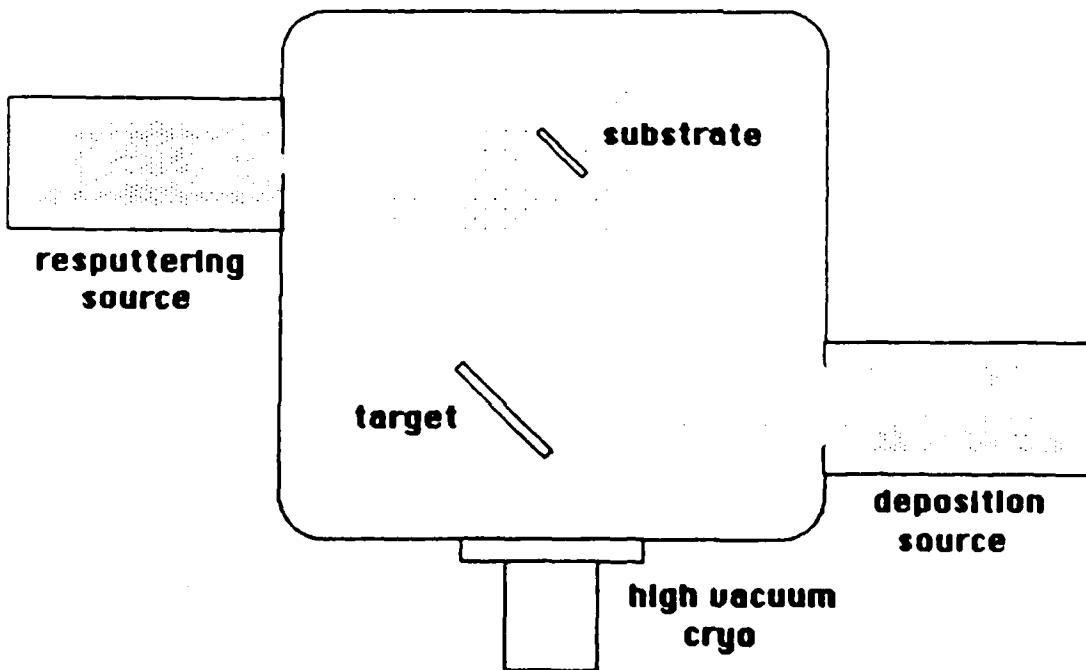
The coercivity ( $H_c$ ) of the ion beam films is generally lower than that of the RF samples.  $H_c$  for the ion beam films ranges from 0.25 to 1.25 Oe, while the RF films have  $H_c$  from 0.25 to 4.0 Oe.  $H_c$  decreases with an increase in film thickness as expected. The data for the ion beam samples show clearer trends with less scatter as can be seen by inspection of Fig. 3. The anisotropy field ( $H_K$ ) is in approximately the same range for both processes (2.5 - 6.5 Oe) as seen in Fig. 3.

Future work involves optimization of the NiFe films' magnetic properties over a wide range of process parameters for the ion beam deposition method. This includes a study of parameters affecting the final

### RF Diode System Schematic



### Ion Beam System Schematic



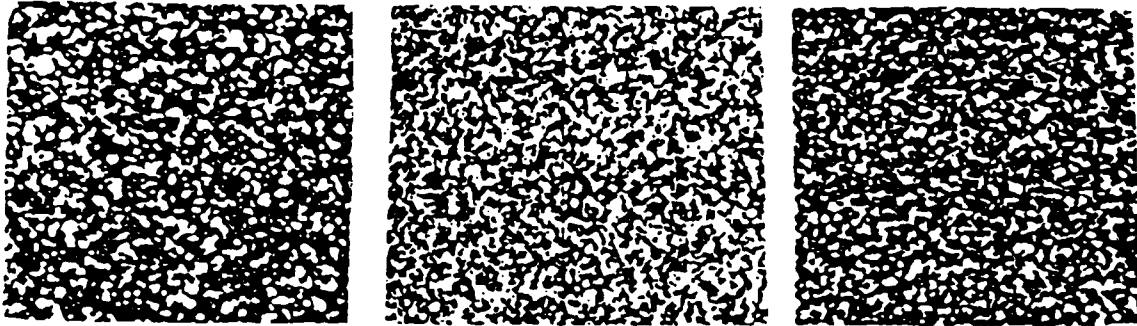


Figure 1: TEM micrographs showing no change in grain size with film thickness. Thicknesses are (l to r) 270, 425, and 680 Å.

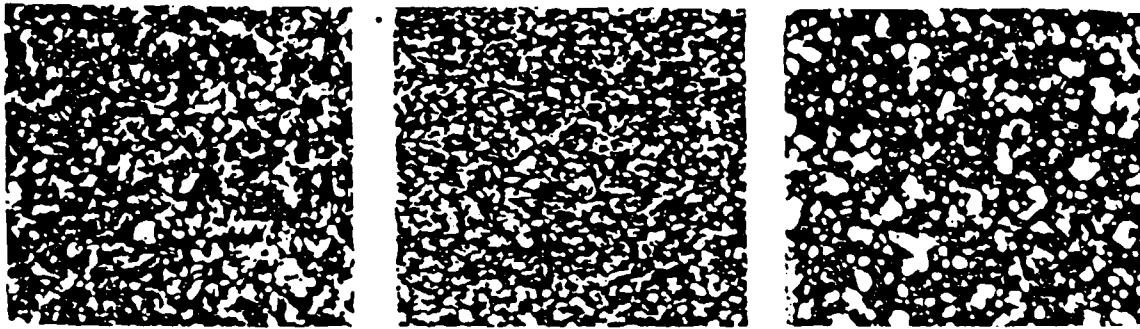
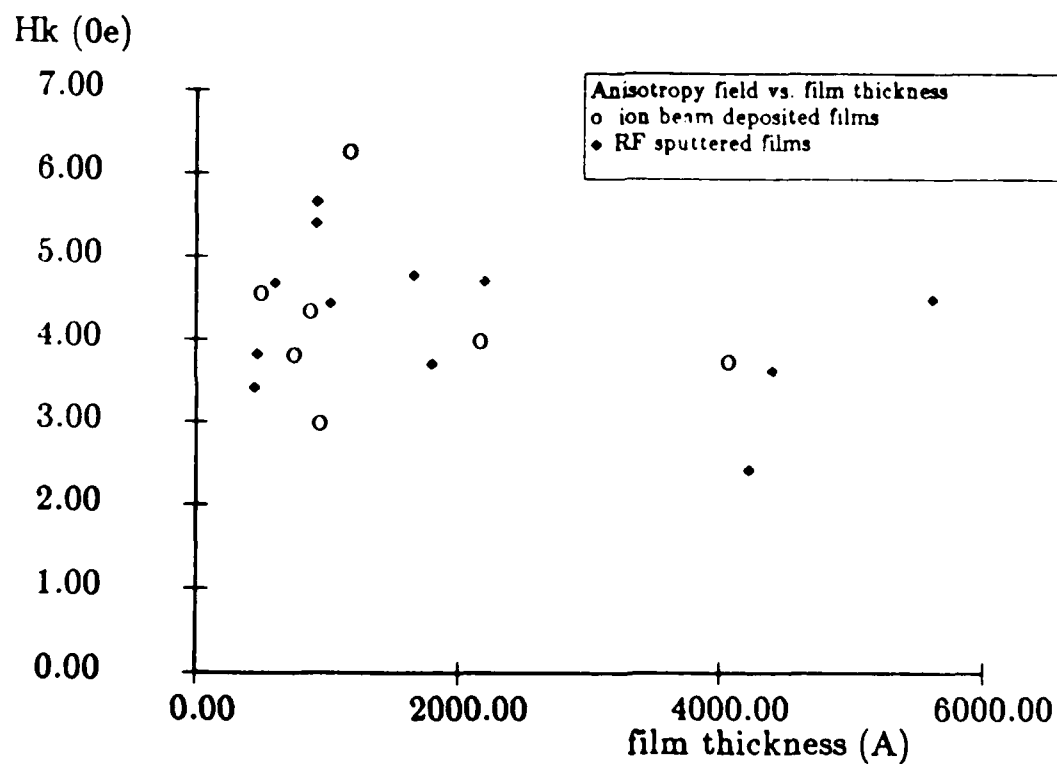
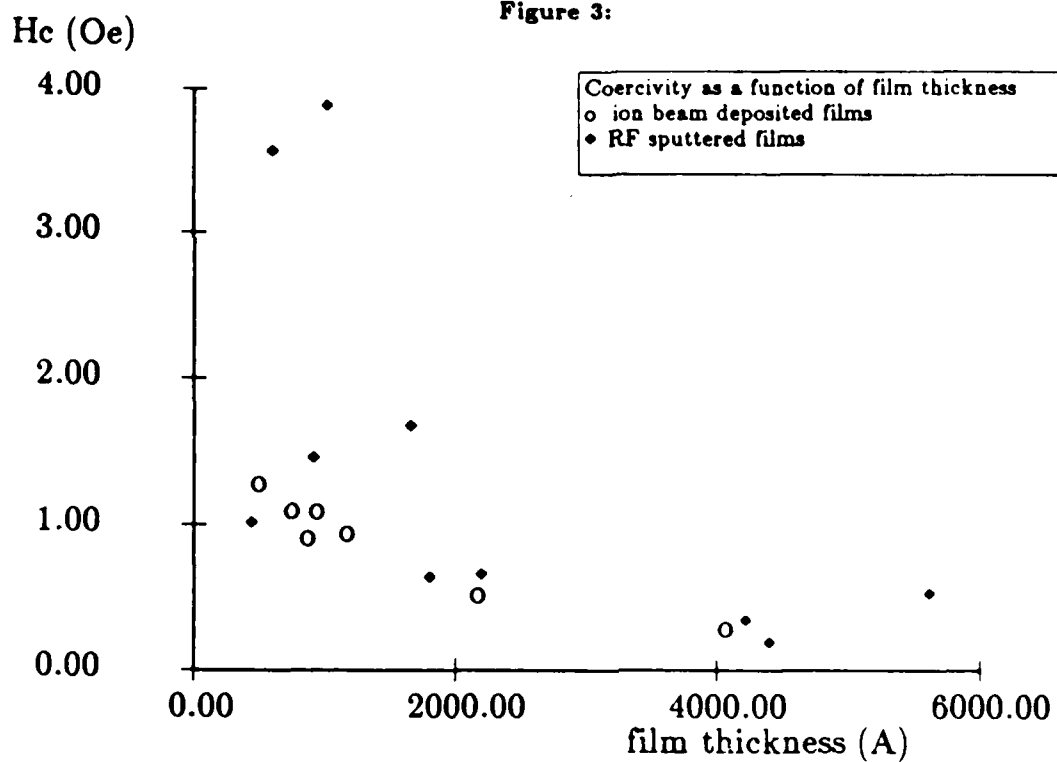


Figure 2: Comparison of grain size for the different processes. RF sputtered film (left) has grains 300-400 Å, ion beam without resputter (center) averages 50-100 Å, and ion beam with resputter (right) measures 100-200 Å.



Figure 3:



composition of the films. Microstructure will be investigated where necessary for explanation of results.

## Magnetic Oxides for Magneto-Optical Recording

### Introduction

For use as a magneto-optic recording medium a material must have a large magneto-optic effect, favorable compensation and/or Curie temperatures, a high room temperature coercivity and uniaxial anisotropy. Meeting with these requirements, oxides which have the advantage of natural stability against corrosion and high resistance to annealing over the commonly used rare earth-transition metal amorphous films are being pursued as candidates for magneto-optic recording applications. The initial phase of this research was a literature search for all available magnetic and magneto-optical data on oxides such as ferrites, magnetoplumbites, hexaferrites, spinels and garnets. After searching out materials exhibiting magnetic parameters favorable to magneto-optic recording, we selected Co-ferrite and bismuth doped garnets as promising candidates because of their high magneto-optic effects and because they had been successfully deposited by sputtering techniques by other groups.

We chose to pursue ion beam deposition as a means of depositing these magnetic oxides because it offers advantages in control over deposition parameters which are not available by conventional sputtering. Ion beam deposition allows isolation of the substrate from the ion generation process and thereby enables more control over substrate temperature, angle of incidence of deposited material and independent control over ion beam current and energy. Deposition can be performed in a lower pressure of argon than conventional sputtering and a second ion gun may be used to controllably resputter from the deposited film during the deposition process. As a result of these differences between ion beam deposition and sputtering, ion beam deposited films have shown many unique characteristics. Films which are deposited onto low temperature substrates generally exhibit better adhesion when ion beam deposited than sputtered. As a result of deposition at lower argon pressures, films can be made with lower argon entrapment by ion beam deposition than by sputtering. As a result of the lower substrate temperatures, grain size in ion beam deposited materials can be smaller than when made by sputtering. This could be important in polycrystalline magneto-optic materials in which media noise is dependent on the grain size. Being able to vary the average angle of incidence of deposited atoms opens the possibility of studying the effects of angle of incidence on anisotropy, microstructure and Kerr rotation. The second gun in the ion beam deposition system offers the possibility of enhancing the mobility of atoms on the surface of the film to allow them to move to local energy minima leading to a more ideal film with fewer defects.

### Properties of Promising Materials

**BiFe Garnets** Because of large magneto-optical effects, Bi-substituted iron garnets are promising as a magneto-optic memory medium. Highly Bi-substituted garnet films on glass and GGG substrates were successfully RF sputtered and characterized by the group of M. Gomi.<sup>1</sup> The films exhibit a uniaxial magnetic anisotropy perpendicular to the film plane. Large Faraday rotation of  $1 \times 10^4$  deg/cm at  $\lambda = 633\text{nm}$ , large  $H_c$  of 600 Oe and remanence-to-saturation ratio of unity indicate its suitability as a magneto-optic memory medium. The films are also suitable for thermomagnetic writing due to the temperature dependence of the coercive force  $H_c$ . Preparation of bismuth iron garnet films by ion beam sputtering onto GGG substrates was reported by T. Okuda.<sup>2</sup> An anomalously large, negative Faraday

rotation was found in the near infra-red region. The value of  $-4.2 \times 10^4$  deg/cm is as large as the highest value reported for LPE grown films. Krumme in an unpublished presentation at the International Colloquium on Magnetic Films and Surfaces in Asilomar in September, 1985, reported the sputter deposition of single crystal bismuth doped garnets, having properties equivalent to LPE grown materials, onto gadolinium gallium garnet substrates. There was speculation that it might be possible to achieve such properties in films deposited onto glass or other non-crystalline substrates if strongly orienting seed layers were first deposited. The group in Philips of Hamburg [P. Hansen *et al.*<sup>3-9</sup>] have studied the magnetic and magneto-optical properties of lead-, praseodymium-, aluminum-, gallium-, bismuth-substituted yttrium or gadolinium(rare earth) iron garnet films which were grown by LPE onto (111)-oriented calcium-, magnesium- or zirconium-substituted gadolinium, samarium or neodymium gallium garnet substrates. Garnet films exhibiting large magneto-optical effects in the visible region and containing  $\text{Pr}^{3+}$ ,  $\text{Bi}^{3+}$  or  $\text{Pb}^{2+}$  ions were described in their published reports. F. Inoue *et al.*<sup>10</sup> in Japan reported that Co, Bi doped garnet films, grown on  $\text{Gd}_3\text{Ga}_5\text{O}_{12}$ (111) wafers from  $\text{Bi}_2\text{O}_3$ -PbO flux, have a large Faraday rotation angle  $\theta_F$  in the visible region. The thermomagnetic writing characteristics of these films were also investigated. Investigation of magnetic properties on iron garnet films were also performed by several workers such as Y. Yokoyama<sup>11</sup> of the Electrotechnical Laboratory in Japan, D.M. Gualtieri<sup>12</sup> of Allied Corporation, H. Takeuchi<sup>13</sup> of Hitachi in Japan and R. Krishnan<sup>14</sup> of the Laboratory of Magnetism in France.

**Co-ferrites** The use of spinel-ferrite in magneto-optic memory applications is creating interest due to its relatively large magneto-optical effects and an optical absorption coefficient suitable for thermomagnetic recording in the visible region. Table 1 summarizes some of these past results. Sputter-deposition of spinel-ferrite films on quartz, glass and stainless steel substrates for magneto-optic memory was published by the group of M. Abe,<sup>15</sup> the  $\text{Co}_{0.6}\text{Cr}_{1.5}\text{Fe}_{0.9}\text{O}_4$  film shows the highest value of  $4.3 \times 10^4$  deg/cm at  $\lambda=633\text{nm}$ . Similarly workers at Philips [J.W.D. Martens *et al.*<sup>16</sup>] have deposited Co-ferrites with large magneto-optic effects. It was shown that the rotation can be increased considerably by optimizing the thickness of the  $\text{CoFe}_2\text{O}_4$  film in combination with auxiliary non-magnetic thin films. At 633 and 780nm it was found that the Faraday rotation was  $0.74 \times 10^4$  and  $6.6 \times 10^4$  deg/cm respectively.

**Ba-ferrites** Ricoh in Japan [H. Machida *et al.*<sup>17</sup>] has recently announced the deposition of Ba-ferrite with good potential for magneto-optic recording applications. Thermomagnetic writing by Ar laser was achieved on the Ba-ferrite perpendicular film. Co-Ti substitution in a Ba-ferrite single crystal produced an enhanced Faraday rotation of  $0.83 \times 10^4$  deg/cm at  $\lambda=780\text{nm}$ .

### Selection of Materials

Based upon the above reports we decided to initially pursue bismuth doped garnets and Co-ferrites. These materials exhibit superior magneto-optic coefficients as compared to Ba-ferrite and have been successfully deposited by sputtering techniques. We believe that good control over film microstructure will be critical to achieving good properties in these crystalline materials and we therefore decided to begin on materials with which others had already had some success. In addition, our extensive experience in the growth and properties of LPE garnets makes us favor garnets as a starting point.

## Sample Preparation and Characterization

The first stage of film fabrication is to optimize the control parameters of the ion beam deposition system in order to deposit the sputtered films.

**Apparatus** The Commonwealth Scientific Corporation dual ion beam sputtering system as shown schematically in Fig. 4 was used to prepare the films. Two Kaufmann ion sources are placed at both sides of the sample chamber to provide controlled ion beams. The Ar ion beam generated in ion gun 1 sputters the target to deposit a film. The ion beam from gun 2 resputters the surface of the deposited film. The substrate holder can be water cooled or heated. Gas flow rate is regulated by a mass flow controller. The incidence angle of the Ar ion beam used to sputter from the target was  $45^\circ$  from the target normal. A neutralizer filament was used to neutralize the positive charge of ions impinging on the specimen stage.

**Target Fabrication** A pressed-powder ceramic disk (6" diam x 1/4" thick) cobalt-ferrite target with the composition  $\text{CoFe}_2\text{O}_4$  bonded onto the copper backing plate was used to deposit the Co-ferrite films in this study.

**Film Deposition** Films were deposited by ion beam sputtering onto glass substrates (1" square, Corning No. 0211) which were washed in a mild detergent, rinsed in deionized water, blown dry with nitrogen and plasma ashed for a minimum of 10 minutes. In our initial films the base pressure was in the low  $10^{-7}$  Torr range. The substrate and target were water-cooled. Substrate angle was kept at 45 degrees in the first few attempts. No bias etching from the second gun was used. Ion accelerating voltage, ion current density and argon partial pressure were controlled and varied from run to run. The sputtering conditions are given in Table 2.

**Film Characterization** The saturation magnetization and perpendicular anisotropy constant were measured with a vibrating sample magnetometer (VSM) and a torque magnetometer respectively at room temperature. A magnetic field of up to 10 kOe was applied. Composition and thickness have not yet been determined, but the films are being forwarded to an industrial laboratory for analysis by Rutherford back scattering.

## Experimental Results

Measurable data were found by VSM and torque magnetometer for films with thicknesses greater than 0.4  $\mu\text{m}$ . No magneto-optic domain patterns could be observed under the microscope. The M-H loop and torque curve showed that the as-deposited films have a magnetically hard axis perpendicular to the film plane with isotropic in-plane magnetization. X-ray diffraction analysis shows that all of the as-sputtered films in the substrate temperature range used in this experiment were amorphous. We have not yet received the results of the film composition analysis. The coercivity was estimated to be less than 200 Oe from high field VSM measurements. Studies of the effects of argon pressure, ion accelerating voltage and ion current density on film characteristics are underway.

## Discussion

Others have reported extremely large perpendicular anisotropy constants in single-crystal cobalt-ferrite films grown epitaxially on MgO substrates.<sup>18</sup> The anisotropy in these films was attributed to strong magnetocrystalline anisotropy, stress induced magnetostrictive anisotropy and shape anisotropy due to

columnar microstructure.

In the initial films grown here, no such strong perpendicular anisotropy has been observed. In fact, comparisons of  $K_u$  to  $-2\pi M_s^2$  indicate no perpendicular component of anisotropy whatsoever. To find the reason for the lack of perpendicular anisotropy, we plan to obtain composition analysis by Rutherford back scattering, and microstructural information from transmission electron microscopy and both x-ray and electron diffraction. We will also experiment with changes in process conditions, including the addition of oxygen to the sputtering gas and the use of oriented substrates.

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Table 1. Reference data on  $\text{CoFe}_2\text{O}_4$ 

| Sample                                     | $M_s(\text{emu}/\text{cm}^3)$ | $K_u(\text{erg}/\text{cm}^3)$ | $H_k(\text{kOe})$ | $H_c(\text{kOe})$ | Ref. |
|--|-------------------------------|-------------------------------|-------------------|-------------------|------|
| $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4$ | 28                            |                               |                   |                   | 18   |
| $\text{Co}_{0.7}\text{Fe}_{2.3}\text{O}_4$ |                               | $8 \times 10^6$               | 35                | 6                 | 18   |
| $\text{CoFe}_2\text{O}_4$                  | 22                            | $4 \times 10^6$               |                   |                   | 18   |
| $\text{Fe}[\text{CoFe}]\text{O}_4$         |                               | $1.8 \times 10^6$             |                   |                   | 19   |
| $\text{CoFe}_2\text{O}_4(\text{bulk})$     | 425                           | $2.0 \times 10^6$             |                   |                   | 20   |

**Table 2. Sputtering Conditions**

|                           |                                 |
|---------------------------|---------------------------------|
| Substrate:                | 0211 Corning Glass              |
| Vacuum: Base Pressure     | $4 \times 10^{-7}$ Torr         |
| Working Pressure          | $(0.9-0.4) \times 10^{-4}$ Torr |
| Gas Flow Rate: Ar         | 8.00 sccm/min                   |
| Ion Accelerating Voltage: | 0.5-1.5 kV                      |
| Ion Current Density:      | 7-15 mA/cm <sup>2</sup>         |
| Ion Neutralizer:          | used                            |
| Substrate Temperature:    | RT                              |

**Table 3. Characteristics of Sputtered CoFe<sub>2</sub>O<sub>4</sub> Films**

| Sample | P <sub>Ar</sub><br>(Torr) | V <sub>accel</sub><br>(kV) | I<br>(mA/cm <sup>2</sup> ) | K <sub>u</sub><br>(ergs/cm <sup>3</sup> ) | 2 $\pi$ M <sub>s</sub> <sup>2</sup><br>(ergs/cm <sup>3</sup> ) | Thickness<br>( $\mu$ m) |
|--------|---------------------------|----------------------------|----------------------------|---|--|-------------------------|
| #.50   | $2 \times 10^{-4}$        | 1.5                        | 10                         | $-0.35 \times 10^5$                       | $0.33 \times 10^5$   | 1.0                     |
| #.51   | $2 \times 10^{-4}$        | 1.0                        | 10                         | $-0.30 \times 10^5$                       | $0.056 \times 10^5$  | 0.6                     |
| #.52   | $2 \times 10^{-4}$        | 1.0                        | 7                          | $-0.93 \times 10^5$                       | $0.37 \times 10^5$   | 0.5                     |
| #.53   | $0.9 \times 10^{-4}$      | 1.0                        | 10                         | $-1.65 \times 10^5$                       | $0.52 \times 10^5$   | 0.6                     |



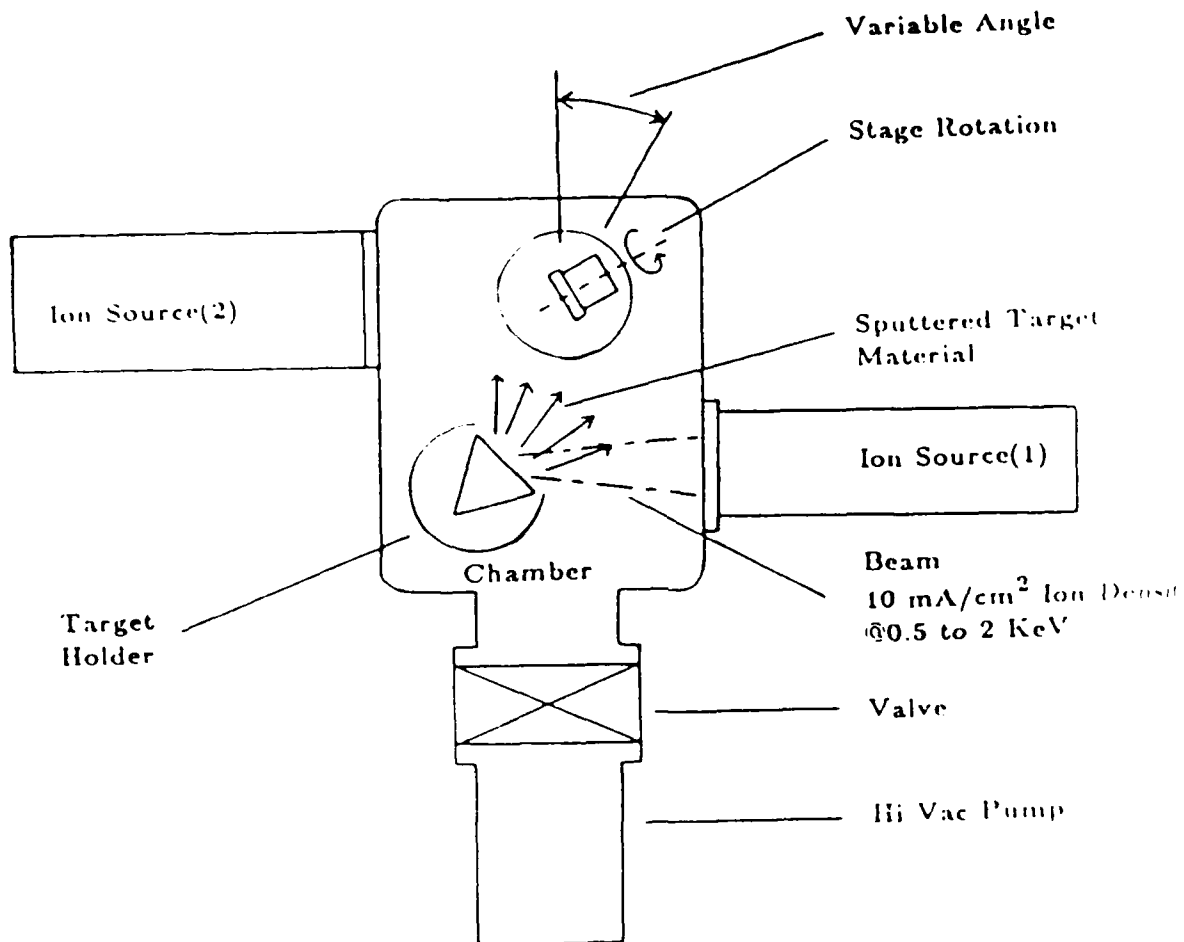


Figure 4: CSC dual ion source deposition system

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